

SULFATE IN SOILS

(An Arizona Method)

SCOPE

1. (a) This test method describes a procedure for determining sulfate content in soil using a turbidimeter. The sulfate content determined is defined in terms of the method and is water leachable or "available" sulfate. Interferences are minimized by use of a reagent blank in the turbidimeter measurements.

(b) This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

(c) See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

(d) The extraction procedure, paragraphs 4 (a) through (d), is the same as is used in Arizona Test Method 736, Chloride In Soils.

APPARATUS

2. Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:

(a) Turbidimeter - with at least $\pm 2\%$ accuracy to 1000 NTU and at least $\pm 5\%$ accuracy to 4000 NTU, with sample cuvettes.

(b) Balances or scales:

1) One balance or scale capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity shall be at least 0.1 gram.

2) An analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M231, except the readability and sensitivity shall be at least 0.1 milligram.

- (c) Graduated cylinder - 100 mL capacity.
- (d) Beaker(s) - 300 mL capacity.
- (e) Magnetic stirrer and stirring bar(s).
- (f) Erlenmeyer flask(s) - 500 mL capacity with stoppers.
- (g) Filtering funnel(s) with filter papers.
- (h) Volumetric Flask - 1000 mL capacity, accurate to 0.3 mL.
- (i) Reagent bottle - 500 mL capacity, with a dispenser capable of dispensing 5 mL of reagent.
- (j) Weighing dish suitable for weighing and transferring less than 0.5 mL of crystalline material.
- (k) Pipettes, Class A - 5 mL capacity, 10 mL capacity, and 25 mL capacity.
- (l) Sample bottle - 200 mL capacity or larger, with cap.
- (m) Dropping bottle.
- (n) Centrifuge, with 50-mL tubes and closures.

REAGENTS

3. (a) Conditioning Reagent. Into a reagent bottle, add 300 mL demineralized water, 30 mL Reagent Grade Concentrated Hydrochloric Acid, 100 mL Reagent Grade Isopropyl Alcohol, 75 grams Reagent Grade Sodium Chloride, and 50 mL Reagent Grade Glycerol. Mix well.

(b) Sulfate Standard Solution, 0.100 mg/mL (100 ppm). Into a 1000 mL volumetric flask, add 147.9 mg Reagent Grade Anhydrous Sodium Sulfate. Fill the flask to the mark with demineralized water. Mix well.

(c) Barium Chloride Solution, 30%. Into a 1000 mL volumetric flask, add 300 grams of Reagent Grade Barium Chloride, Dihydrate, FW 244.28, Crystals, 20-30 mesh. Fill the flask to the mark with demineralized water. Mix well.

(d) Nitric Acid, 20%. Measure 10 mL concentrated nitric acid into a clean beaker containing approximately 40 mL demineralized water and mix well. Transfer to a dropping bottle. **Caution must be exercised in preparing and using this solution! It should be properly labelled and treated as a hazardous material.**

PROCEDURE

4. (a) Weigh 100.0 ± 0.1 grams of soil passing a No. 10 sieve into a 500 mL Erlenmeyer flask.

(b) Weigh 300.0 ± 0.1 grams demineralized water into the flask.

(c) Stopper the flask, shake vigorously, and let the mixture stand for one hour.

(d) Filter the extract into a sample bottle obtaining 125 to 150 mL of filtrate and put a cap on the bottle.

(e) The filtrate must be clear. If it is not clear, transfer a portion of the filtrate to a centrifuge tube(s), put a cap on the tube(s), and centrifuge to clarify the filtrate. If centrifugate is still not clear, add 2 drops of 20% nitric acid and repeat.

(f) Pipette a 25 mL aliquot of Sulfate Standard Solution into a 100 mL graduated cylinder. Record the size of the standard aliquot, in mL, as "ALSTD".

(g) Dispense 5 mL of Conditioning Reagent into the cylinder. Fill the cylinder to the 100 mL mark with demineralized water as accurately as possible.

(h) Mix and transfer the mixture to a beaker by pouring back and forth. Place a stirring bar into the beaker, place the beaker onto a stirrer, and stir until thoroughly mixed.

(i) Transfer some of the mixture to a cuvette filling the cuvette to the mark, insert the cuvette into the turbidimeter and adjust the turbidimeter to read 100.0% transmittance.

(j) Remove the cuvette and adjust the turbidimeter to read 0.0% transmittance.

(k) Repeat the turbidimeter adjustments in steps (i) and (j) until stable readings are obtained.

(l) Transfer the mixture in the cuvette back to the beaker, pipette 5 mL of Barium Chloride Solution into the beaker, place the beaker onto the stirrer, and stir the mixture for 5 to 7 minutes to allow for development of any precipitate.

(m) Mix and transfer some of the mixture containing the precipitate back into the same cuvette, by pouring back and forth, filling the cuvette to its mark. Insert the cuvette into the turbidimeter and read the absorbance.

(n) Record the absorbance value as "ABSTD" to the nearest 0.001 absorbance unit.

(o) Pipette a 25 mL aliquot of filtrate from the sample bottle (or centrifuge tube) into a 100 mL graduated cylinder. Record the size of the sample aliquot, in mL, as "ALSAM".

(p) Repeat steps (g) through (m). Record the absorbance value as "ABSAM" to the nearest 0.001 absorbance unit.

(q) Compare the values "ABSTD" and "ABSAM". If "ABSAM" is larger than "ABSTD", pipette a suitably smaller aliquot (for example, 10 mL) of filtrate from the sample bottle (or centrifuge tube) into a 100 mL graduated cylinder. Record the size of the reduced sample aliquot, in mL, as "ALSAM". Repeat steps (g) through (m). Record the absorbance value for the reduced sample aliquot as "ABSAM" to the nearest 0.001 absorbance unit. Compare the values "ABSTD" and "ABSAM" for the reduced sample aliquot. If the resultant "ABSAM" is still larger than "ABSTD", repeat using yet a smaller sample aliquot (for example, 5 mL) of filtrate.

CALCULATION AND REPORT

5. (a) Calculate sulfate content in the soil in parts per million, "S", and report to the nearest 10 ppm as follows:

$$S = 300 \times \left[\frac{ALSTD \times ABSAM}{ALSAM \times ABSTD} \right]$$

Where: ALSTD = Size of the standard aliquot, 25 mL.
ABSTD = Absorbance corresponding to ALSTD.
ALSAM = Size of the sample aliquot, mL.
ABSAM = Absorbance corresponding to ALSAM.